Study of the Manufacturing Parameters Affect the Fabrication of Nano and Micro Composites

Hebatalrahman, A

Consultant in materials sciences and materials applications, Egypt
hebatalrahman11@yahoo.com hebatalrahman@naseej.com

Abstract: In this research the fabrication quality parameters of micro and nano composites will be mentioned. The manufacturing parameter such as temperature, pressure, and cooling rate were studied. Poly methyl metha acrylate reinforced by fiber glass was considered as case study in the current work. The effect of fiber size (length to diameter ratio) and fiber volume fraction were evaluated. The fabrication temperature of the composite was evaluated. The factors affecting the heating rate such as power, volt and furnace efficiency were studied. At the end of research, the results and discussions explain the main parameters affecting fabrication of nano and micro composites.

[Hebatalrahman, A. **Study of the Manufacturing Parameters Affect the Fabrication of Nano and Micro Composites].** Researcher. 2012;4(1):15-23]. (ISSN: 1553-9865). http://www.sciencepub.net.

Key words: Nano, Composite, thermoplastics, temperature, pressure, cooling rate, manufacturing, techniques

1. Introduction:

Polymer nanocomposite have attracted great attention due to the unique properties introduced by nanofillers, which typically refer to carbon blacks, silicas, clays or carbon nanotubes (CNT). The polymer matrix acts as a supporting medium and the improvement in the properties of nanocomposites generally originates from the nature of these nanofillers[(1),(2)]. The term reinforcement is normally used to denote the increase in rigidity and strength achieved by dispersing inorganic fibers or particulates fillers in the polymer matrix [(3): (7)].

Effectiveness of fiber in the composite

The effectiveness of fibers or reinforcements in the composite depends mainly on three factors [(8), (9)]. **First**, it must have good bond strength between the polymer matrix and the fibers, this is usually achieved by coating the fiber with a material which bonds well to both fiber and polymers [(10)] **Second,** the effect of fiber concentration on the properties of the composites [(11) :(16)].**Third,** The effect of fiber size in the other words, the effect of length to diameter ratio [(17): (20)].

In general, the fibers are assumed to be homogeneous, much stiffer than the polymer matrix, regular spaced, perfectly aligned or randomly distributed. The polymer matrix is assumed to be homogeneous and isotropic linear elastic [(21), (22)]. The interface between the fiber and the polymer matrix is an anisotropic transition region which must provide a stable bond between them [(23), (24)].

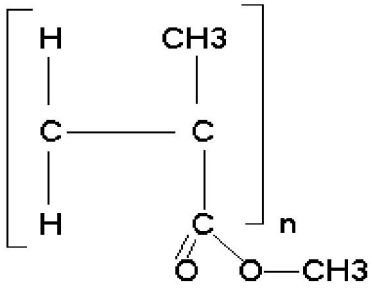
Solvent casting, melt mixing and coagulation methods are the common fabrication methods for nano-composites, some researches use combined methods, such as solvent casting in conjunction with sonication followed by melt mixing [(25),(26)]. For example the combination of solvent casting and melt mixing for SWNT/PMMA composites with considerable improvement in nano-tube dispersion [(27), (28)]. PMMA was dissolved in organic solvent that was also used to disperse additives and after that it cast into a dish. In situ polymerization method has also been used to make nano-tube-based nano-composites starting with nano-tube and monomers [(29), (30)]. The most common in situ polymerization methods involve epoxy in which the resin (monomer) and hardeners are combined with additives or reinforcement prior to curing (polymerization) [(31),(32)]. Its resin can be cross-linked after mixing with hardener [(33), (34)].

The development of nanotechnology is closely related to the new economy and high technology products towards 21st century [(35)]. As a new material, the nano-particle has certainly caught the imaginations of researchers around the world. Although the production technologies of nano-particles have had significant progress in the past few years, the commercial applications of nano-particles are often limited by the cost, handling, and safety issues [(36), (37)]. To address these application problems, a proprietary mechanic-chemical bonding technology has been developed to fabricate nano-composite powders. It can create nano-scale multifunctional composite materials contributing to the development of advanced materials and devices for rechargeable batteries, fuel cells, ceramics, metals, superconductors, cosmetics and pharmaceuticals. The technology is an enabling technique to broaden the applications of nano-particles. In these work, new method was developed, the nano materials can be bonded together using mechanical energy without any binders in a dry process [(37)].

2. Experimental work

I. Materials

The matrix is polymethyl metha acrylate (PMMA. It is thermoplastic prepared from monomer (methyl methacrylate) by additional polymerization process; polymer was in the granule form with 5mm diameter.



Fiberglass (brand milled fibers) are glass filaments coated with a specific sizing to enhance resin compatibility and hammer milled to a specified bulk density. Fibers are hammered milled to an average fiber length. Average fiber length is determined by the input glass and process conditions. In the current work, milled fibers are made from "E" (electrical) glass which conforms to MIL-R- 60346C specification with average bulk density 0.55g/cm^3 in floccular and powder form with length to diameter ratio (L/d=50 and, L/d=1) respectively.

II. Preparation of Material

Grinding

A blinder with variable speeds is used to change the pieces of thermo-plastics into powder form, The blinder speed is about 15,500 rpm, work at 200 volt - 50 Hz, and the grinding time is about 90 sec. Switch off for cooling is lasted about 180 sec to prevent the agglomeration of the particles and achieve reasonable degree of quality in grinding. The retained granules after sieving will be returned to blinder.

Mixing

Automatic mixing for thermoplastic in the powder form with short fibers at different aspects ratio was done. The amount of material required was calculated by changing the volume fraction percent into weight percent (L/d=50 and, L/d=1) respectively.

Composite material preparation

A methodology for design of new composite material is based on strength to weight ratio, manufacturability, and cost requirements are presented. Simple method for manufacturability and cost elements are developed and combined to produce a new technique for manufacturing of thermoplastic based nano-composites. This work establish very small setup for production of the thermoplastic based composites, this small unit become more suitable for determination of the best fabrication parameters at certain composition fig (1) show the experimental setup for manufacturing of nano-composites a) fabrication test machine and b) automatic blinder (mixing and grinding). The manufacturing quality parameters were determined as temperature, pressure, cooling rate and holding time (time of application of force).

The material was prepared by grinding and mixing of thermoplastic in the powder form. The mixing of fiber with thermoplastics and other modifiers was done. The process is done with certain volume fractions depend on the properties required in the final product. Equation number (1) and equation number (2) show the role of mixture to indicate respectively the density and the weight , the composite material was prepared in the solid form, it had different characteristics depends on the fiber percentages (volume fraction of fibers) and fiber size (length to diameter ratio).

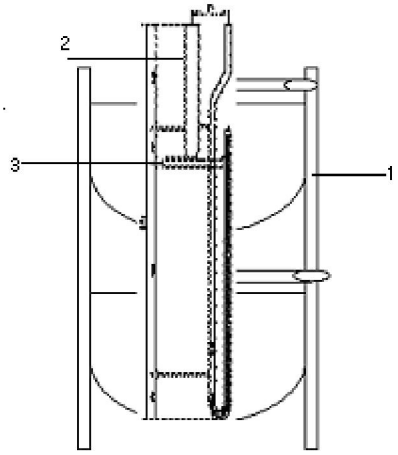
$$\rho_c = \rho_f V_f + \rho_m V_m \qquad (1)$$

$$W_c = W_f V_f + W_m V_m \qquad (2)$$

Where
V_f = volume fraction of fiber
V_m = volume fraction of matrix
ρ_f = density of fiber
ρ_m = density of PMMA
W_f = weight percent of fiber
W_m = weight percent of PMMA

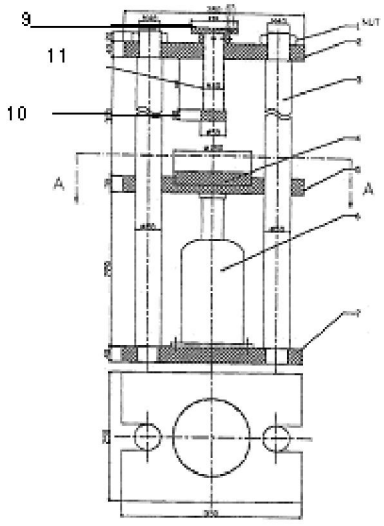
Fabrication tests

The factors affect fabrication of nano-composites were determined by the following fabrication tests, all factors were fixed while only one factor changed. The fabrication conditions of the polymer matrix were (T= 240ºC, P= 160 kg / cm^2) so this values were considered as indication values in the experimental tests. The tests were divided into four categories. First, the effect of cooling rates. Second, the effect of fabrication temperature. Third, the effect of the fabrication pressure. Fourth, the effect of fabrication time. Fig (2) show the manufacturing parameters which affect the fabrication of polymer based composites. Fig (3) shows The manufacturing steps of nano-composites materials. Table (1), (2), (3) and (4) show the fabrication tests.



b) automatic blinder (mixing and grinding)

1. fixation element
2. mixing chamber
3. sieving



a) fabrication test machine

1. Nuts for fixation
2. Fixed arm of the pressing
3. Upper part of mold
4. Lower part of mold
5. fixed rod
6. pressing body
7. Lower bed
8. pressing part
9. manometer
10. thermocouple
11. stop watch

Fig (1) Experimental set up used in fabrication of nano-composites

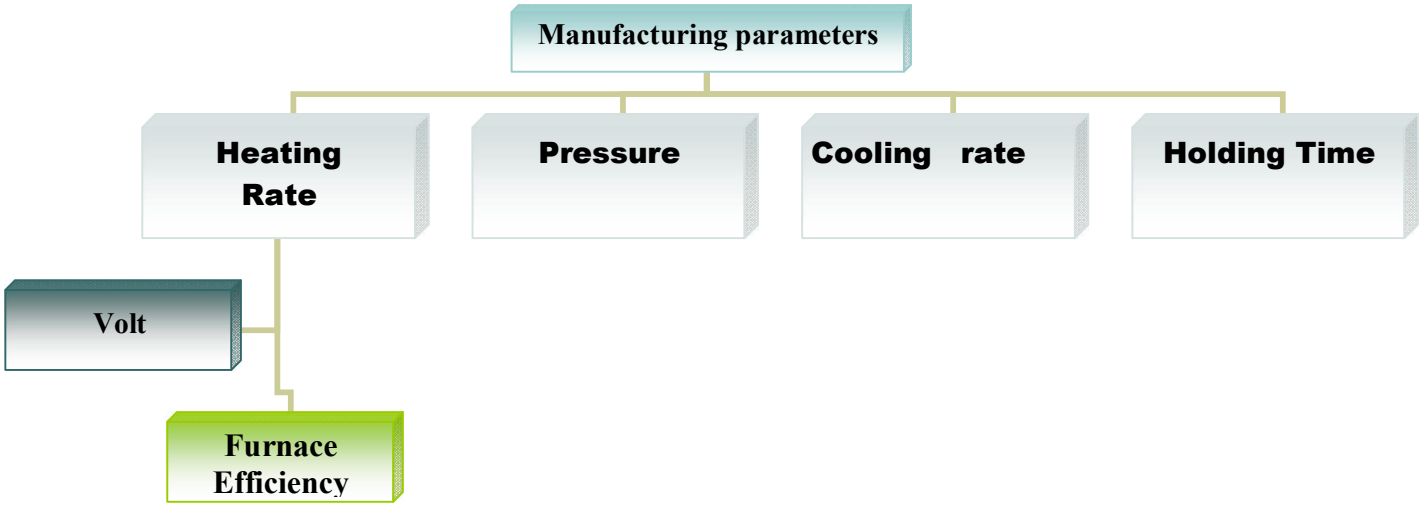


Fig (2) schematic diagram of the manufacturing parameters

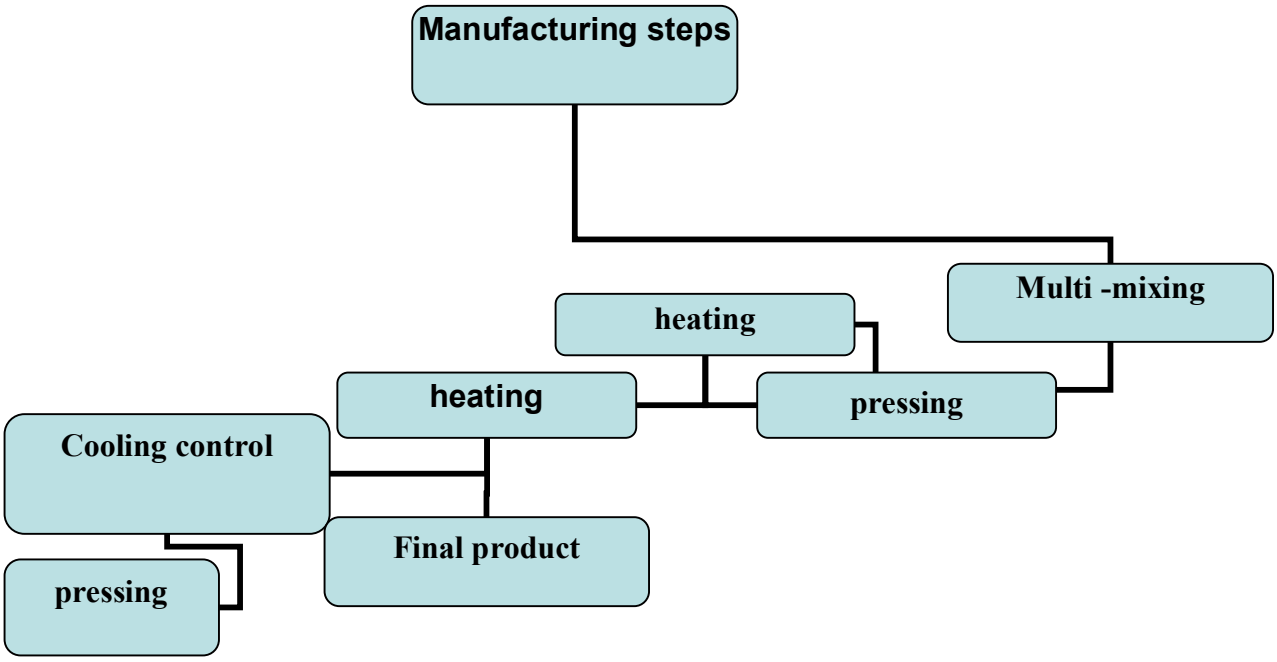


Fig (3) The manufacturing steps of nano-composites materials

Test1

The suitable cooling rate:

The aim of these test was the selection of the best cooling rate, table (1) show the steps of the test to determined the cooling rate, the test was done at the following cooling rate ranges-:

1- slow cooling rate mold cool (M.C)
2- medium cooling rate air cool (A.C)
3- fast cooling rate water cool (W.C)

Table (1) the effect of cooling rates

Factors affecting manufacturing	Temperature °C	Pressure N/m^2*10^5	Time seconds	Cooling rate
Values recorded during experimental work	300	160	300	M.C
	300	160	300	A.C
	300	160	300	W.C
states	constant	constant	constant	variable

Test2

The effect of temperature:

The aim of this test was the selection of the fabrication temperature at each volume fraction and fiber size, table (2) shows the steps of the test to determine the temperature of fabrication. The test was done at the following temperatures (340°C, 300°C, 240°C, 200°C, 140°C).

Table (2) the effect of fabrication temperature

Factors affecting manufacturing	Temperature °C	Pressure N/m^2*10^5	Time seconds	Cooling rate
Values recorded during experimental work	340	160	300	W . C
	300	160	300	W . C
	240	160	300	W . C
	200	160	300	W . C
	140	160	300	W . C
states	variable	constant	constant	constant

Test3

The effect of the fabrication pressure

The aim of these test was the selection of the fabrication pressure at each volume fraction and fiber size, table(2) show the steps of the test to determined the pressure of fabrication The test was done at the following pressures 50 **N/m^2*10^5**,100 **N/m^2*10^5**,160 **N/m^2*10^5**, 300 **N/m^2*10^5**, 500 **N/m^2*10^5**

Table (3) the effect of the fabrication pressure

Factors affecting manufacturing	**Temperature ºC**	**Pressure N/m^2*10^5**	**Time seconds**	**Cooling rate**
Values recorded during experimental work	240	50	300	W . C
	240	100	300	W . C
	240	160	300	W . C
	240	300	300	W . C
	240	500	300	W . C
states	constant	variable	constant	constant

Test4

The effect of time of application of force

The aim of this test was the selection of the minimum time required for fabrication time at each volume fraction and fiber size, table (4) shows the steps of the test to determine the time of fabrication. The test was done at the following desecrate times 30s, 60s, 90s, 120s, 150s.

All tests at different test conditions were repeated at different volume fractions of fibers 0%, 17%,35%,50%,65%, and at different fiber sizes (length to diameter ratio) (L/D=50 and L/D=1), the average of three reading were recorded.

Table (4) the effect of time of application of force

Factors affecting manufacturing	**Temperature ºC**	**Pressure N/m^2*10^5**	**Time seconds**	**Cooling rate**
Values recorded during experimental work	240	160	30	W. C
	240	160	60	W. C
	240	160	90	W. C
	240	160	120	W. C
	240	160	150	W. C
states	constant	constant	variable	constant

3. Results & Discussions

The suitable cooling rate is indicated from the first test, this test was done under constant temperature, pressure and fabrication time. Both air cool and furnace cool were failed, the samples were pores. The fast cooling rate (water cool) gave the best results. The same results were repeated at all volume fractions and all fiber sizes (length to diameter ratio). The cooling rate is a function of the matrix type, it independent of the amount of reinforcement and its size.

Temperature is the most important and the most effective factor in the fabrication of plastic based composites. The temperature increase when the amount of energy consumed increase the best fabrication temperature in the range from 300ºC to 350ºC, when the temperature increase the dissociation of the matrix occur, when the temperature decrease the fabrication quality also decreased. The temperature range is a strong function of the matrix, there is no significant changes can be recorded at any aspects ratio or at any concentration of fibers. Fig (4) show the heating rate as a function of the voltage change at constant current, While fig (5) show the variation of temperature with time during furnace heating , the heating rate and the time required to reach required temperature were depend on the furnace efficiency.

The effect of pressure was studied at constant temperature and holding time. The reading of the monometer was recorded at every volume fraction and aspect ratio. There was a significant increase in the fabrication pressure with the increase of the volume fraction of fibers fig (6) show The variation of fabrication pressure with volume fraction of fibers at different aspects ratio. The pressure required for

fabrication was increased when the aspects ratio decreased, the decrease in fiber sizes need more force to get red of micro voids and pores. The decrease in fiber size from L/D=50 to L/D=1 lead to increase in the fabrication pressure about 50 MN/Cm$^{2.}$

The effect of fabrication time was studied at constant temperature and pressure. At relatively low concentration of fiber volume fraction in the range from 0% to 35% the holding time was constant at 240s at all aspects ratio considered in these work. At higher fiber concentrations in the range from 50% to 65%, The holding time increase with the decrease in the aspects ratio, the holding time increase about 60s to 120s when the fiber size dropped from L/D=50 to L/D=1 Fig(7) show the variation of fiber volume fraction and fiber size with holding time during fabrication. The holding time was the less significant factor affect the fabrication of composites.

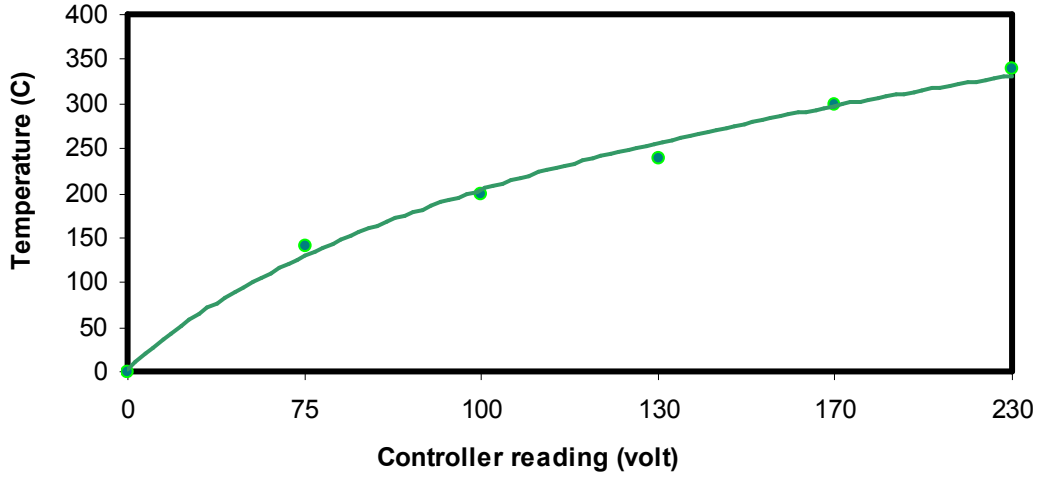
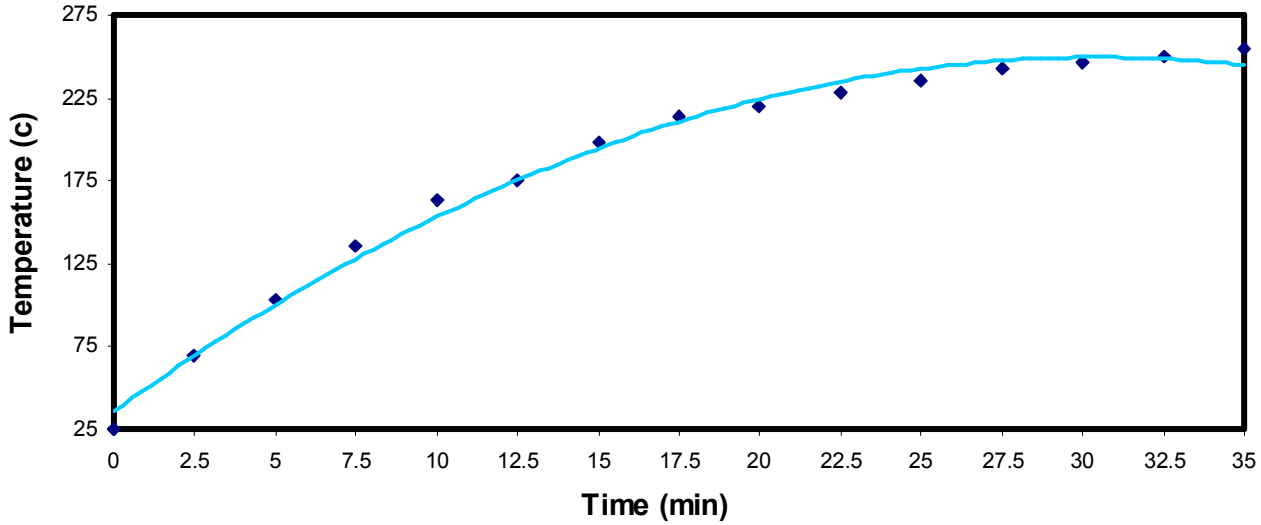
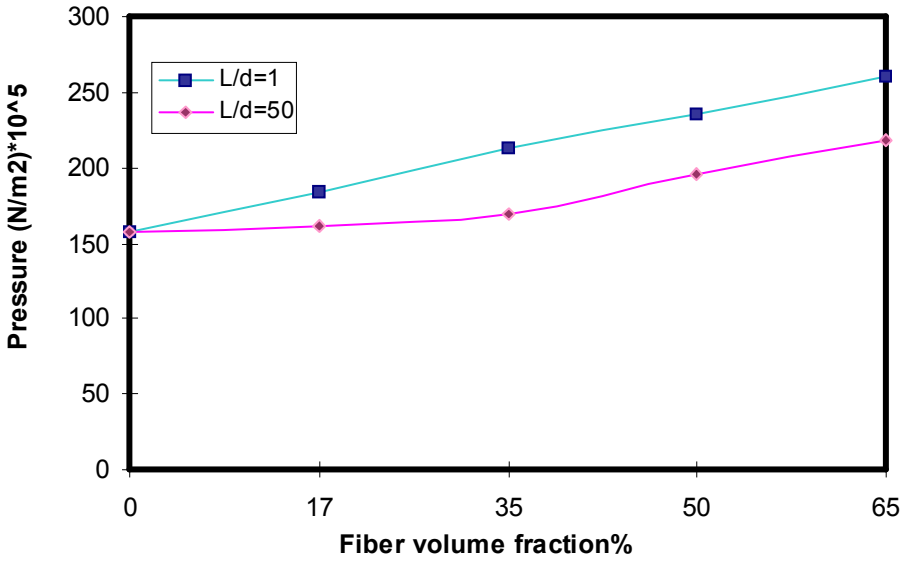


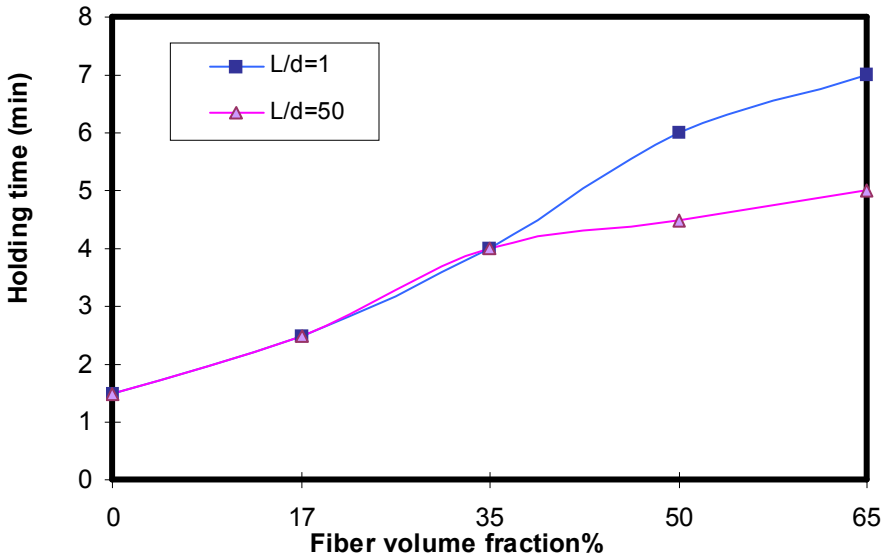
Fig (4)Heating rate control



Fig(5) The variation of temperature with time during furnace heating



Fig(6) The variation of fabrication pressure with volume fraction of fibers at different aspects ratio



Fig(7) The variation of fiber volume fraction and fiber size with holding time during fabrication

Conclusions

1. the main factors affecting the fabrication of nano-composites powders in the current work were temperature, pressure, cooling rate and holding time
2. The optimum condition in fabrication of (PMMA) polymer is considered as indication value in fabrication of composite at different volume fraction and aspects ratio.
3. Temperature is the most important and the most effective factor in the fabrication of plastic based composites
4. The fast cooling rate (water cool) was the optimum cooling rate at every length to diameter ratio at all concentrations of fibers.
5. Pressure required for fabrication was changed with both volume fraction of fibers and fiber size (length to diameter ratio) to produce non pores specimen
6. The holding time was the less significant factor affect the fabrication of composites, the effect of these factors appeared slightly at fiber concentrations more than 35%.
7. The fabrication method and furnace efficiency were important factors in the fabrication quality of thermoplastic based composites.
8. both cooling rate and temperature are factors related to matrix type regardless of the reinforcement type, size and volume fraction.
9. The new technique developed in these work was unique and suitable for a wide range of thermoplastic based, thermoset based and metal based nano and micro composites regardless of the reinforcement or filler type.

The new technique was registered as patent in the Egyptian patent office.

Corresponding author
Hebatalrahman, A
Consultant in materials sciences and materials applications, Egypt
hebatalrahman11@yahoo.com
hebatalrahman@naseej.com

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